IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant:

A. Stoyanov et al.

Attorney Docket No.: 25384

Application No.: 10/815,206

Weverhaeuser Legal

Art Unit: 1731 / Confirmation No: 9520

Filed:

March 31, 2004

Examiner: D.R. Cordray

Title:

BLEACHED CROSSLINKED CELLULOSIC FIBERS WITH HIGH COLOR

AND BRIGHTNESS

DECLARATION OF KATHY A. WELCH PURSUANT TO 37 C.F.R. § 1.131

Seattle, Washington 98101

April 13, 2006

TO THE COMMISSIONER FOR PATENTS:

- I, Kathy A. Welch, declare as follows:
- 1. I am employed by Weyerhaeuser Company as a Scientist.
- 2 I have read and am familiar with U.S. Patent Application No. 10/815,206, (the 206 application).
- 3. I have read and am familiar with U.S. Patent Application Publication No. US 2003/0208859 A1 ("the Neogi reference") that published November 13, 2003.
- 4. Prior to the publication date of the Neogi reference, I conducted pilot line trials in which cellulose fibers were crosslinked with a crosslinking agent in the presence of a polyol and then bleached. These bleached crosslinked fibers were prepared under the direction of Angel Stoyanov, M.Sc., an inventor of the subject matter claimed in the '206 application. following describes two pilot line runs Trial T-75 and Trial 82 performed on September 8, 2003 and October 28, 2003 respectively, that provided cellulosic fibers crosslinked with citric acid in the presence of sorbitol described in Table 4 of the '206 application.
 - In Trial 75, southern pine kraft pulp (CF416) was crosslinked with an impregnation solution including citric acid (crosslinking agent), sodium hypophosphite (SHP, crosslinking catalyst), and sorbitol as set forth on page 1 of my laboratory

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notebook (Exhibit A attached to this declaration). The crosslinked fibers were then treated with a bleaching agent that was either hydrogen peroxide or a combination of hydrogen peroxide and sodium peroxide. The target bleaching conditions for the trial, Samples B, H, L and N in Table 4 of the 206 application are set forth on the same page of my laboratory notebook. The corresponding notebook entries are in the Run Matrix on page 1 in EXHIBIT A are listed under the Run ID as C, D, H and I, respectively. Unbleached control samples represented by samples A, K and M in Table 4 correspond to Run ID B, G and J, respectively, in the Run Matrix on page 1. The treated pulp was separated into individualized fibers and cured to provide citric acid crosslinked fibers.

- b. For each run, nine (9) samples were taken in duplicate. Sample B in Table 4 is represented by C-1 to C-9, page 22 and 23; sample H by D1 to D-9, page 23; sample L by H, page 24; and sample N by I, page 24 and 25 (see Exhibit B). Unbleached control samples A, K, and M in Table 4 (which correspond to Run ID B, G and J, respectively, in the Run Matrix on page1), are represented by BB-1 to BB-9, page 22; G, page 24 and J, page 25, respectively. Each run was sampled at the baler feed and the samples were taken at approximately two minute intervals during each run.
- c. Hunter L, a, and b values were measured. The results were tabulated as set forth on pages cited in paragraph c., above.
- d. In Trial 82, southern pine kraft pulp (CF416) was crosslinked with an impregnation solution including citric acid (crosslinking agent), sodium hypophosphite (SHP, crosslinking catalyst), and sorbitol as set forth on page 1 of my laboratory notebook (Exhibit C attached to this declaration). The citric acid crosslinked fibers were treated with a bleaching agent that was either hydrogen peroxide or a combination of hydrogen peroxide and sodium peroxide. The target bleaching conditions for the trial,

Samples I and J in Table 4 are set forth on page 83 of my laboratory notebook. The corresponding notebook entries are under the Run ID as E and F, respectfully in the Run Matrix. Unbleached control samples previously mentioned in paragraph b., above.

- e. For each run, five (5) samples were taken. Sample I is represented by E, page 92 and sample J by F, page 92. Each run was sampled at the baler feed and the samples were taken at approximately two minute intervals during each run.
- f. Hunter L, a, and b values were measured. The results were tabulated as set forth on pages 92, **EXHIBIT D**.
- 5. All of the bleached crosslinked cellulose fibers described above were prepared prior to November 13, 2003. In accordance with accepted Patent Office practice, the dates in my laboratory notebook pages presented in **EXHIBITS A-D** have been redacted.
- 6. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Date: 4/13/06

Mathy A. Welch

Kathy A. Welch

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Project No. 142 - 4506
Bock No. 14615 TITLE ARPL

Page No. 23)(Sample	g Viriali	Semple		Sampling	Tool Days	and the second second second	Zav
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				1 b		evoevos © o everage	52,98-95,64 1.34-2,0.62,98,60-1.26 52,82-96,67-1.38 679, 86,63-4,28	6.79
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essed & Unde	75 BF 75 BF			1 a	09/09/03 0	9/08/03 0 9/08/03 0	79.34 94.88 1.56 8.32 96.00 -1.51 79.67 94.95 -1.50 8.24 98.09 -1.45	8.42
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Project No. 142 -4506 ARRL Trial 75 (conx.) Book No. 14615 25 From Page No.24 Kals 75. 91.03 95.26 4.48 7.61 9631 4.38 7 67 00/00/03 DEMONITOR: 6.79 98.484-1.13 6.81 BF. 09/09/03 08/08/03 0. 7.27 88.40 -1.33 7.31 82.00 95.4Z BF 09/09/03 82.22 95.44 6.93. 96.45 82.50° 85.57° 6.80 96.55 6.81 6.87: 96.50 08/09/03 9/09/03 8.38 95.78 -1.38 8.58 08/08/03 29/09/03 78.53 94.64 8.63 95.82 8.77 78.65, 94.82 79.53, 94.82 77.84, 94.70 BF 08/08/03 5.00 0 80:31: 95.21 8.08 **36.27** 96.16 08/06/03 91:40: 95.35 09/09/03 **81.76** 95.36 700 96 36 BF: 00/08/03 ø 96.15 B O. 80.17 94.06 00/00/03 10 70) 95.20 F 8F 00/00/03 2.09 BF BF 09/09/03 09/09/03 8.72 00/08/03 96.07 **9.13** 3.23 850 09/09/03 874 9.07 09/09/03 1.64 8.91 98.50 90.37. 41.21 19 96.44 4.19 07 96.30 -1.44 52 98.35 -1.35 8.15 7.5% To Page No. 26 Witnessed & Understood by me, Date nvented by Date Recorded by

253 924 3253 EXHIBIT C Project No. 142-4506 APRI Trial 82 Determine OSIO Cire Time Book No. 1465 83 prion Page No. and Temperature to Hit SK target of O.O. and Huser L APPL THAT T-82 Kari Project Manager: Augul Sloyanov. Project Number: 00050 W532 615 874 731 142-4504 Objective: Deten e the interaction of cure time and temperature leading to a cross-link product with 5k and Hinter b values of 0:15 and 5, or less for both, respectively. Safety: Normal safety processions taken with operation of the APPL unita-Read MSDS for Sorbitot and handle per MSDS recommendations. Run Conditions: Pulp Type: Pulp Sheet Emen Food Ran CF416 Hammermall Noscher Cape Hammermall Roser Tap Spec Instruction Solutions Con-0.095 inches l6,493 fpm CS10 Control H 62% solid Services 1/51% series: Adjuste 1:0 / 2 per rise County and the Intercematica Solu Target Hammersell Rend Consistence 619Z Target Chemical on ODCF Pulp CS10 Control - 7.6% Citie Acat, 9.883% SHP & Causio Serbitol - 6.0% Citrio Acat: 1.5% Section & 0.75% SHP (all as 100% Punity). Impreguation Solution Ra Nominal Core. Temperatu Nominal Core Time: 360.TF See Rim Matrix Target Product Moist anoisturization Solution eriodisturization Rotanacker Settling 60% of scale (20 psi water & 28 psi air) ttrix. Cure H) Targets Conditions Solution Region 20.0 bs. of Cure Tim ilis. NaOH mts H₂0 MADMIT 44.7 0.0 0.0 0.0 0.0 44.7 44.7 5.0 629.1 44.5 44.5 44.6 0.011 629.1 You Samples: Pulp Peet Rolle: Sample Analyses Pula Reof Rolls للب بعو ما Moisture Hales Feede mill Reed Moisture, Brightness, Hunter and CIE Color (U, 7 de 14 days), 5K and Odior $\gamma \omega$ Fish Feed Rolls and Hammermill Feed moisture complet will be placed in 9612 inch morphs bags. Baier Feed moisture samples will be placed in 9° x 12° bags. Baier Feed FA() Analysis, Highiness and color samples and 9% samples will be placed in 13° x 18° bags. (States bags since we will need quite a bit of sample for these analyses)). Chier samples will be placed in glass bottles. Angel will sample, bottles, Take on Samples #2 + 44. Impregnation Chemical Solution: CS10 Control Solution Chemical Solution Recipe Sorbitol Solution Solution Make-up Contingency Factor 27 4 26 Target Solution Component Weight in Pounds eric Acid (as-received), lbs. 25.25 20.1 itol (as-received), lbs: 0.00 7.1 SHP (as-received), ibs 2.73 3.0 0.0 0.60 tic (as-received), ibs 208.25 208.2 238.6 238 R Total lbs 25.0 1.05 25.0 Volume of Water, gallons: 1.0 impregnation Solution Specific Gravity: e of impregnation Solution, gallons: 27.1 27.2 1.13 2 Witnessed & Understood by me; Date 2.07 oll Xail Recorded by 7/a

04-19-2006

EXHIBIT D

	Project No Book No	: 142-45 : 146 -5	DG TITLE	ACCLL	Tess	l 82 (ر بسر	72		
ge No. 91		ing Variable Sen		Sampling Test		tanes (. b [*	(46)		
	Tital Poin 82 BF 82 BF	A A	vence Pad Replicate	Dele Date 10/28/03: 10/28/0 10/28/03: 10/28/0	0	76.43 83.98 -1.0 76.28 93.95 -1.4	1: 9.33 95.29 8: 9.39 95.27	1.46 9.54 4.43 6.6 °		
	82° EFF 82° EFF		2 f a	10/28/03 10/28/0 10/28/03 10/28/0			# 9.92 94.93 6 10.59 94.7	4.45 8.57 -†.4 ;f0.19 -1.5 ;10.94		
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	82 BF	4	4 1/2 2	10/28/03 10/28/0	ा वरवा शुरू छो ∉ 0 =	75.87 S1.58 -4.1	1 9.27 94.19 2 9.71 94.71			
	82/ BF		5 1 e	10/28/03 10/28/0 10/28/03 10/28/0	90 0 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	74.43 83.33 -4.3 74.56 83.421.	1: 10.01: 94.75: 3: 10.04: 94.85:	1.27 10.30 1.26 10.53		
	82 BF	8	6 - 1 - b: 1-::	10/28/03 10/28/0 10/28/03 10/28/0	3 C	MAR 91A4 43 78.15 95.37 -1.9	9 10 16 94.87 4 9.14 96.39	1.23 - 16.44 1.88 (r. 8.29 - 1)		
	82 8F	9 B	1(* 1) . b*. 2	10/28/03 10/28/0 10/28/09 10/28/0	average /	78.87 95.37 - 2.0 78.81 95.37 -1.9 83.02 96.19 -1.8	9.25 M.39 -	1.95 0.91 1.92 8.40 1.74 1.3	370	
	62 AF	8	27. hi b. 3 17 b.	10/28/03 10/28/0 10/28/03 10/28/0	-	69.15 95.71 82.86 85.93 4.9 81.24 95.84 1.9	1. 7.63 M.ES	1.95 - 8.07 1.96 - 7.65 1.85 - 8.27		
	82, BF	Ø	3, 1 6	10/28/03 10/28/0 10/28/03 10/28/0	3: 0 merage	\$1.34 95.69 1 81.29 95.67 -13 81.7 95.62		JS 127		
	82 BF 82 BF	8 - B	6 15 6	10/28/03 10/28/03	3 0 ·	667 9561 -17 9126 9572 -17	8 8.25, 96.58 - 8.04 96.66 -			
	82 BF 82 BF	- B		10/28/03 10/28/0 10/28/05 10/28/0	9 0 everage	80.69 95.64 1.9 80.49 95.67 -1.9 80.58 95.68 -1.9) 8.55 98.63; - P 8.46 95.62 -	87 8.85 LBT 9.55		
	82 SF 82 SF	, c	U TO TO B	10/28/03 10/28/03 10/28/03 10/28/03			(* 6.59; 97.01; /- (* 7.12) 96.56 * 6.86 96.78; -	ALL DANGE TO THE ENGLISH OF THE SECOND		
	82 BF 82 BF	G C	2 1 B	10/28/03 10/28/03 10/28/03 10/28/03			8: 6.87: 97:18 7: 7:51: 97:05 7: 7:19: 87:12	<i>8</i> 6, 7.53		
	82 BF 82 BF	. c	1 1 0 0 12 1 10 10	10/28/03 10/28/03 10/28/03 10/28/03		62.54 98.09 12 61.95 95.67 1.9 62.25 65.96 1.9	+ 7.62 96.96 - + 7.67 98.78 -	SF : 7.71		
	82) BF # 82 BF	i č		10/28/03 10/28/03 10/28/03 10/28/03		8252 96.01 -2.0 8255 -96.04 -196 8258 -88.85 -4.56	7.49 96.89 - 7.59 96.92 -	第4.7年 80.77年		
	824 BF 82 BF	C S		10/28/03 10/28/03 10/28/03 10/28/03	12 O	84.51°, 95.82°, -1.5 82.31°, 95.92°, -1.64	7.98 96.75 7.53 96.62 -	85 8.048 76 7.56		
	82) BF 82 BF	D; 1		10/28/03 10/28/03 10/28/03 10/28/03	X - 0	81.00 95.00 -1.00 81.00 95.00 -1.00 81.0 95.00 -1.00	7.91 96.62 -1 k 7.51 96.67 -1	57 7.68 58 7.55		
	82° a . 98° 82° a . 98°	D 2	1 w	10/28/03 10/28/03 10/28/03 10/28/03		利用 報酬 4の 配格 第79 19 配行 65年 15	7.2 96.71	15 7.22		
	82 SF 82 SF	D/*. 3		10/26/03 10/26/03 10/26/03 10/26/03	L - 07)	60.29 95.67 -4.54 63.13 95.91 -4.42 62.88 95.92 -1.56	6.86" 96.81% - 1	36 6.67		
	82 BF	D 4	1 .	10/28/03 10/28/03 10/28/03 10/28/03	**************************************	82.54 95.92 4.59 82.5 95.84 1.62 82.52 95.71 1.68	7.85 96.82 -1 7.27 96.76 -1			
	82 BF	D. 5		10/28/03 10/28/03 10/28/03 10/28/03	greerage (**	62.41 95.76 -4.65 84.13 96.05 -1.45	7.24 96.71 -1	391 8.3		
	82 EF	D 6		10/26/03	0	0625 98.06 -1.32 06.78 08.33 -1.2	614 56.89 -1 464 97.14 -1	32 6.11 15 4.56		
व	82 BF	E 2		10/28/03 10/28/03 10/28/03 10/28/03	T 0 1 2 3	65.34 . 95.96 - † 38	4.68 97.29 -1 5.31 96.87 -1	15 4.54 35 5.27		
	82 GF 82 GF	E 2	1 8	10/28/03 10/28/03 10/28/03 10/28/03	aicide.		4.74, 96.67 4.683 96.87 -1 5.22, 97.24 -1	27- 4.00		
Tav.	. 82 BF		The state of the s	10/28/03 10/28/03 10/28/03 10/28/03	o o	MART 98.32 -1.34	5.32 97.14 -1 5.32 97.19 -1	74 6.27 27 6.27		
1	82 BF 82 BF	F .	† b:	10/28/03 10/28/03	average 🕍	16.79 16.50 -1.25 16.75 96.63 -1.25 16.77 16.61 -1.27	6.07 97.37 -4.	22 5.01		
*	82 BF 82 BF	E 5	1 . 6	10/28/03 10/28/03 10/28/03 10/28/03	O (95.85 96.3 -1.29 B.M. 96.28 -1.28	5.28 57.18 -1.	24 5.27 22 6.23		
	82 BF 82 BF		1 6	10/28/03 10/28/03 10/28/03 10/28/03	0 0 everage	667 963 125 662 9627 13 668 9629 429	47, 97,12 47,17, 97,1 -1. 4,71, 97,11 -1.	2 4.84 25 4.65 23 4.66		The state of the s
	82. BF 82. BF	F 2 F 2	1 6	10/28/03 10/28/03 10/28/03 10/28/03	0	9625 9639 - 13 9657 9637 - 125	5.07 97.15 -1. 4.78 97.12 -1.	25 5.02 18 4.74		
	82 7 BF 821 BF	FEET 3	1 b	10/28/03 10/28/03 10/28/03 10/28/03	0 1	MA 16 98 32 -1 32 15.88 98 17 1 3	5 13 97 14 -1. 5 09 97 02 -1.	27 5.08 25 5.04	20	Parties 1
e & Understood b	4 47 18 20 20 20 10 10 10 10 10 10 10 10 10 10 10 10 10			10/28/03 10/28/03 10/28/03 10/28/03	8 0		4.94 / 97.15 -1. 5.5,28 : 97.17 -1.	29 4.88 34 5.23	A Communication of the Communi	
	62 9F 62 9F	F 3		10/28/03 10/28/03 10/28/03 10/28/03	average: 4	M.25 M.25 M.27	5.11 97.16 -1.	12 5.66 29 5.15		
GE 24/24 * RCVD AT 4/19/2		15.00			average	738300 * CSID	527 97.21 -1. 253 924 3253 *	57 5.29 BS 5.22	s):32-20	

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